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Exhibit A

LABORATORY NOTEBOOK

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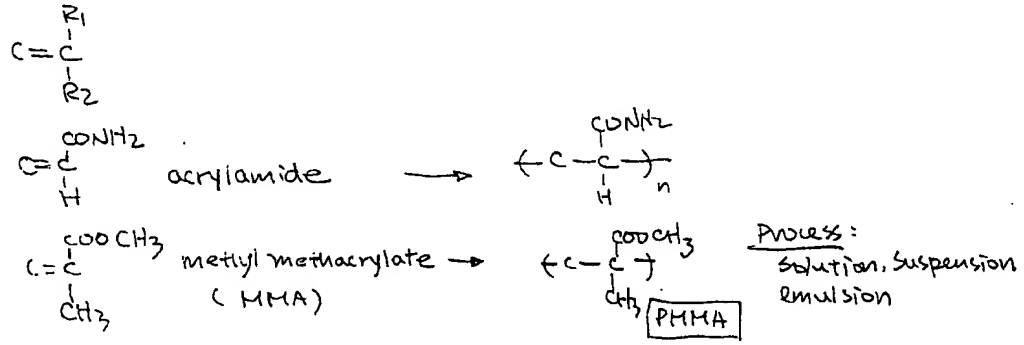
Rm 105 Staaffer II

acrylic family (Principles of polymerization G. Odian 3rd ed. Wiley Interscience)

(P.311)



Process: solution, suspension emulsion

R_1	COOH	COOH	CN	Cl
R_2	H	CH_3	H	Cl
name	poly(acrylic acid) water soluble	poly(methacrylic acid) "	polyacrylonitrile	poly(vinylidene chloride)

R_1 $-OCOCH_3$

R_2 $-H$

name poly(vinyl acetate)

water based paint

{ radical
{ ionic polymerization

chain polymerization

monomer: C=C (vinyl species) → $\oplus C-C\ominus$, $\bullet C-C\bullet$

or $-\overset{O}{\overset{\|}{C}}-$ (aldehyde, ketone) → $-\overset{:O\ominus}{\overset{|}{C}}-$ $\oplus$

type of chain polymerization undergone by various unsat. monomers

monomer	type of initiation: radical	cationic	anionic	
ethylene	+	−	+	
~~vinyl ester~~, acrylate, methyl acrylate, acrylonitrile methacrylonitrile.	+	+	−	electron withdrawn group. eg $C=C^{\delta-}$ + $\oplus$ → $C=C^{\delta+}$...
styrene, methylstyrene	+	+	+	

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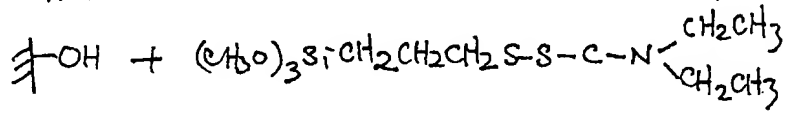
Ref: L Liang, X. Feng, J. Liu P.C. Rieke, G.E. Fryxell Macromolecules 1998 31 7845–50.

Initiator:



N,N'(diethylamino)dithiocarbamoyl propyl(trimethoxy)silane

DATMS

Ref 15, 16.

+

NIPAAm / BisAAm

UV. 254 nm

Reaction

reflux

48h 56°C

250ml 3neck flask

12g CPTMS + 4g SDDC + 100ml acetone

3-chloropropyltrimethoxysilane, 97% Aldrich → purified reduced distillation.

$(CH_3O)_3Si\,CH_2CH_2CH_2-Cl$. (43568-6)

or

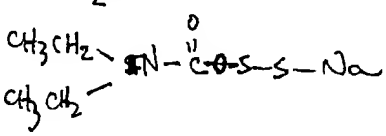
3-chloropropyl triethoxysilane 95% Aldrich (10-2)

L0089 7037 SIC 24070.

SDDC:

Sodium N,N'-diethyl dithiocarbamate

(



[Carbamate $NH_2-C(=O)-OR$] [dithiocarbamate $NH_2-C(=S)-SR$]

CP: sodium diethyl dithio carbamate trihydrate

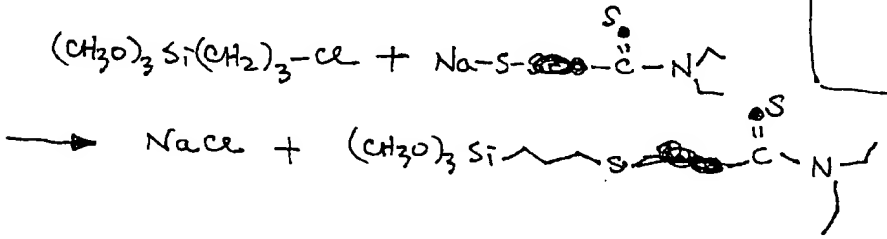
$(C_2H_5)_2N-C(=S)-S-Na\cdot 3H_2O$

FW = 225.31

mp = 95–98.5°

hygroscopic. 22868-0 5g 17.55

Reaction:



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Redo surface polymerization.

$\frac{1000}{75} = \frac{1}{0.075} = 13.$

polyacrylamide $+C-\underset{\underset{C=O}{|}\\ NH_2}{C}+_n$.

acrylamide $CH_2=\underset{|}{C}-\overset{O}{\overset{||}{C}}-NH_2$. MW 71

0.75 g $\frac{0.75g}{71} \sim 0.01$ mol.

0.01 mol acrylamide / 0.75 ml.

2,2' dipyridyl 0.0369 MW 156.19 $(2.3\times10^{-4}$ mol$) = \frac{0.01\text{ mol}}{0.75\times10^{-3}\text{ l}} = 13$ M too high!

Cu(I)Cl 0.0067 g 98.99 $(6.8\times10^{-4}$ mol$)$

DMF 8ml 0.75

should reverse

Calc. ( ) (Wang, 1995)

Cu(I)Cl : bipy = 1 : 3 ($\sim 10^{-4}$ mol) M_0 : Cu(I)Cl

$= 10^{-4} : 3\times10^{-4}$ (mol) $= 100 : 1$

$= 0.01\text{ g} : 3\times156.19\times10^{-4}$ $= 10^{-2}$ mol : 10^{-4} mol

$= 0.01\text{ g} : 0.04685\text{ g}$ $= 0.71 : 0.01$ g

Rough estimation

What thickness means in this case

Assumption: density is const = bulk material

$d_4^{20} = 1.189$ For PAC 50% wt in H_2O.

$1.189 = \frac{1+x}{2} \Rightarrow d_{PAC} \sim 1.37$

$\left(\text{needed mole of monomers}\right) = \frac{Ah\,d(\text{total weight})}{71} = \frac{1\text{ cm}^2\times1.37}{71} h(\text{cm}) = 0.019\times10^{-7} h(\text{nm})$

$\cong 2\times10^{-9} h(\text{nm})$ moles.

According to this, we can greatly reduce the amt of monomers from original recipe

ex: $h = 10^4$ nm ($= 10\ \mu$m)

$\Rightarrow$ needed monomers $= 2\times10^{-5}$ mol

Assume densely packed · 1 molecule / nm^2

For $A = 1\text{ cm}^2 = 10^{14}$ nm $\Rightarrow 10^{14}$ # PAC $= \frac{10^{14}}{6\times10^{23}} = 1.7\times10^{-10}$ m $\sim 2\times10^{-10}$ m

DP $= 2\times10^{-5}$ mol $/ 2\times10^{-10} = 10^5$ $10^5\times0.5 = 5\times10^4$ nm $\sim O(10\ \mu$m)

(good agreement!)

For DMF 1 ml. make 0.01 M acrylamide soln $= 0.01\text{ mol}\times71 = 0.71$ g

$0.01 = \frac{x\text{ mol}}{10^{-3}\text{ l}} \Rightarrow x = 10^{-5}$ mol. $10^{-5}\text{ mol}\times71 = 0.00071$ g

Roughly for DMF 10 ml acrylamide 0.0071 g $\Rightarrow \frac{0.001}{0.01} = 10^{-2}$ M:

Better soln: 0.1 M $\Rightarrow$ 10 ml DMF + 0.071 g acrylamide good enough for cover the whole surface.

$CH_2=\underset{|}{C}-\overset{O}{\overset{||}{C}}-NH_2$ MW = 71.1

$CH_2=\underset{|}{C}-\overset{O}{\overset{||}{C}}-NH-\underset{|}{CH}-CH_2-CH_2-CH_2-NH_2\cdot HCl$ MW = 178.7

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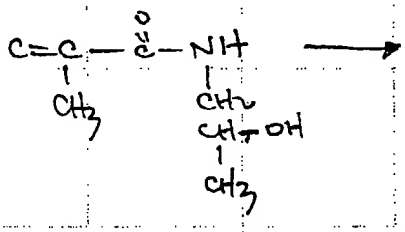
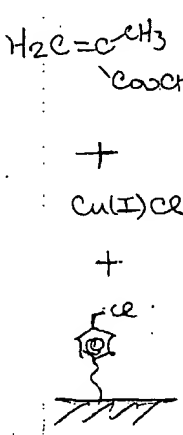
Objective: Repeat protocol exp 7. For N(2-hydroxy propyl) ~~meth~~ methacrylamide.



HJV6d N-(2-hydroxypropyl)methacrylamide [21442-01-3] MW 143.2 mp=67° 10g 121.25



FW=130.14 d=1.073

HEMA in 10ml DMF 0.1mol = 13g HEMA

+

Cu(I)Cl(bipyridyl)$_2$ Cu(I)Cl (MW=99) 0.23g (0.0023 mol) 1

+ bipyridyl (MW=156.19) 1.08g (0.0069 mol) 3

new materials.

chlorophenyl propyl trimethoxysilane 3min 1% in acetone.

+

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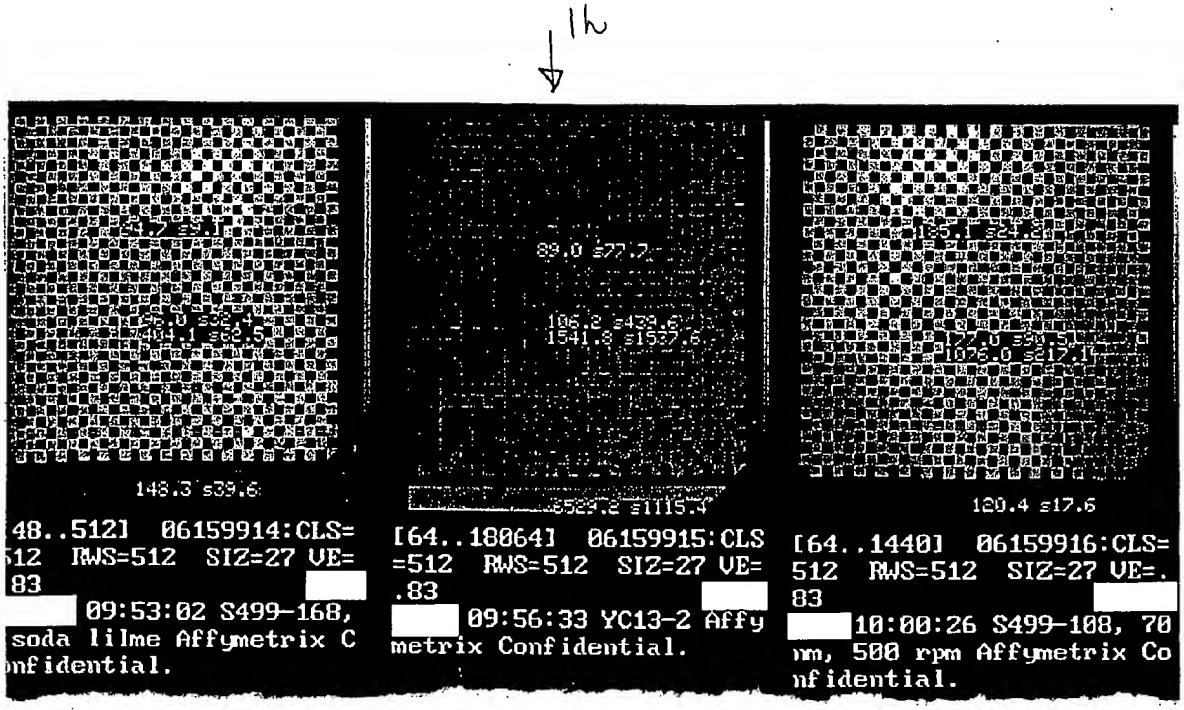
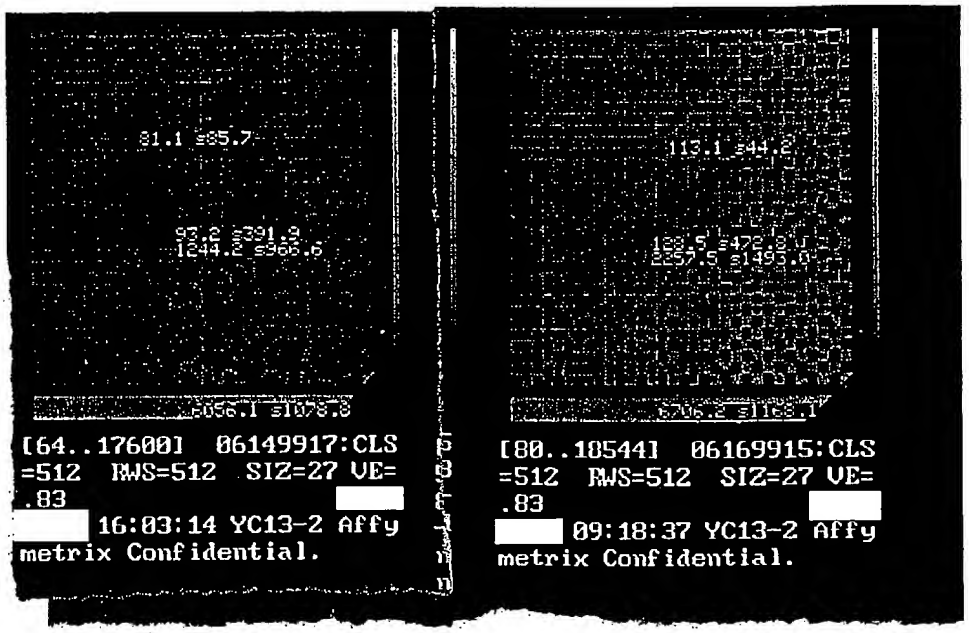

1h
148.3 s39.6
48..512] 06159914:CLS=
512 RWS=512 SIZ=27 VE=
83
09:53:02 S499-168,
soda lilme Affymetrix C
nfidential.
89.0 s77.2
106.2 s439.6
1541.8 s1537.6
[64..18064] 06159915:CLS
=512 RWS=512 SIZ=27 VE=
.83
09:56:33 YC13-2 Affy
metrix Confidential.
120.4 s17.6
[64..1440] 06159916:CLS=
512 RWS=512 SIZ=27 VE=.
83
10:00:26 S499-108, 70
nm, 500 rpm Affymetrix Co
nfidential.


81.1 s85.7
1244.2 s968.6
[64..17600] 06149917:CLS
=512 RWS=512 SIZ=27 VE=
.83
16:03:14 YC13-2 Affy
metrix Confidential.
113.1 s44.2
[80..18544] 06169915:CLS
=512 RWS=512 SIZ=27 VE=
.83
09:18:37 YC13-2 Affy
metrix Confidential.
17h
40h

Subject: Exp 13 Scale up · exp 11

Objective: AIBN — HEMA.

glass — 10 pieces. Si substrates — 4 pieces.

Comment: last exp (exp 11) the thickness only reaches up to 10 nm. I think it's due to the low surface coverage of AIBN layer (I forgot to work it in darkness, so the final thickness checked by Si wafer sample is only half of the thickness of exp 10)

Improvement: This time I will work under dark and high concen of initiator layer.

Sample	Initiator Condition ( )	HEMA rxn.
13-1	glass + AIBN/toluene dark, Et_3N, rm temp in N_2 glove bag. starting 4:00 pm ~	work on condition e10-3 · 20% HEMA/DMF 65°C 24h. degass. freeze thaw 3 times HEMA 40 mL DMF 160 mL
13-2		
13-3	"	
13-4	"	
13-5	"	
13-6	"	store in darkness.
13-7	"	
13-8	"	

DNA strands on pattern → deprotection EtOH/EDA ethylene diamine (1/1) 4h → w/ DI H_2O → hybridization 50 μM. DNA target — tag in 6XSSPE soln → (1h) shake rm temp. → ↓ 17 hr. (hyb) → scan 10 am 1st scan @ → ↓ >4 hr (total 40h) (hyb) → 2nd scan → ~~not scan~~

another protocol: 45°C 16h.

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hyl

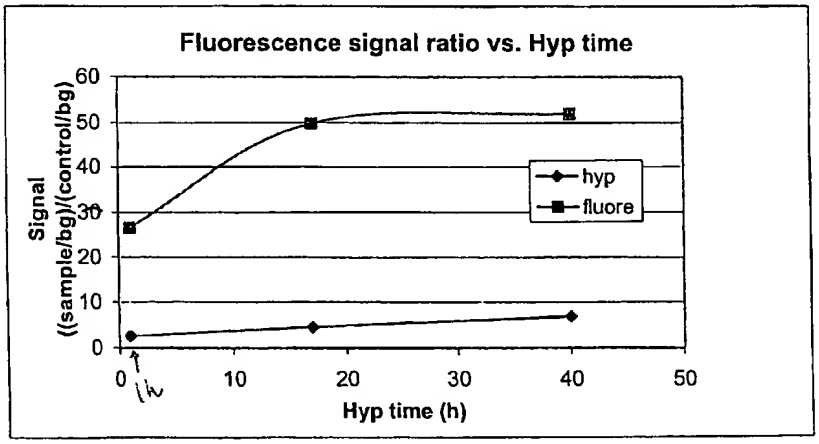
sample	hyp time(h)	hyp	backgd	fluorescein	hyp/bg	fluo/bg
e13-2	1	1244	88	6056	14.1	68.8
control 1	1					
control 2	1	362	66	171	5.5	2.6
e13-2/control		3	1	35	3	27
		1244/362	88/66	6056/171	14.1/55	68.8/2.6

sample	hyp time(h)	hyp	backgd	fluorescein	hyp/bg	fluore/bg
e13-2	17	1542	89	6529	17.3	73.4
control 1	17	339	101	140	3.4	1.4
control 2	17	404	95	148	4.3	1.6
e13-2/control		4	1	45	5	50

sample	hyp time(h)	hyp	backgd	fluorescein	hyp/bg	fluore/bg
e13-2	40	2258	113	6706.2	20.0	59.3
control 1	40	225	94	99	2.4	1.1
control 2	40	318	93	114	3.4	1.2
e13-2/control		8	1	63	7	52

experiment 13
report on file: e13-fluor.xls (thin's chang)
pHEMA initiated by AIBN
Fluorescence measured by Marc Glazer
Experiment condition: AIBN20h)- pHEMA(20h 65c) - DNA probes-hybridization
estimated pHEMA thickness: 5 nm (estimated by Si(100) ellipsometry; need further confirmed by XPS)

Comments for improvement: high standard deviation(ca 1500) -- surface film is not uniform enough -- ne
long hybridization time



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Subject.....

	rxn condition
13-9	" (same as previous)
13-10	"
13-11	same batch but Si(100)
13-12	Si(100)
13-13	"
13-14	"

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